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COMPLETE SPECIFICATION.

Process for the Production of Distributable and Non-caking Fertilisers containing Calcium Nitrate.

We, LONZA ELEKTIZITÄTWERKE UND CHEMISCHE FABRIKEN AKTIENGESELLSCHAFT, a Swiss Company, of Aeschenvorstadt 72, Basle, Switzerland, and 5 EMIL LÜSCHER, of Eichenstrasse 25, and ERNST STIRNEMANN, of Gartenstrasse 105, both in Basle, Switzerland, both of Swiss Nationality, do hereby declare the nature of this invention and in what 10 manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to a process for the production of distributable and non- 15 caking fertilisers containing calcium nitrate.

The calcium nitrate commercially obtainable contains substantial amounts of water and has the property of 20 deliquescing rapidly in the air. Anhydrous calcium nitrate has always hitherto been obtained in a very fine mealy form and is considered, in general, as even more hygroscopic than the ordinary 25 water-containing calcium nitrate. There has, therefore, been proposed a method for producing from unsuitable calcined calcium nitrate a distributable product of small hygroscopicity by inoculating the 30 calcined salt during its cooling with calcium nitrate containing water. The calcined calcium nitrate obtained in this way, on account of its high melting point, cannot be brought into the granular form 35 advantageous for fertilising purposes by the usual process, for example by spraying.

According to the present invention a readily distributable and non-caking pro- 40 duct containing calcium nitrate especially suitable for fertilising is obtained by evaporating a calcium nitrate solution to a sandy-pasty consistency in which it contains about 90—95% of calcium 45 nitrate (calculated as anhydrous calcium nitrate) and then converting the concentrated product without any substantial further concentration into granular form by mechanical disaggregation in bulk at 50 a temperature substantially above atmospheric of the order of 50° to 100° C.

The process claimed of mechanical disaggregation at elevated temperatures

of the sandy-pasty mass, of which the calcium nitrate at a concentration of 55 about 90—95% consists, excludes fundamentally a spraying process, since the mass is not liquid enough for this purpose.

By proceeding according to the present 60 invention it has been unexpectedly found that the apparently non coherent product obtained in this way, in spite of its sandy appearance, rapidly sets on cooling to very hard granules which are much 65 more non-caking and distributable than any calcium nitrate fertilisers previously known in commerce. This is especially the case with the calcium nitrate fertilisers containing ammonium nitrate 70 hitherto so very susceptible to caking. It has further been ascertained that the product obtained according to the present invention deliquesces much less readily in the air and that the granules themselves 75 on taking up the same amount of moisture, retain the compact non-caking form which is not the case with ordinary calcium nitrate.

The evaporation process for preparation 80 of the 90—95 per cent. calcium nitrate product, preferably in its end phase, may be carried out in vacuo in a kneading machine adapted to be heated and 85 cooled.

It has further been found that the material being treated can also be mixed before or during the cooling with other 90 fertilisers without detrimentally affecting the desired properties, indicated above, of the product to be obtained. The material which is being treated can be mixed, for example, with potash salts such as potas- 95 sium nitrate, potassium chloride or potassium phosphate or mixtures of these. The preparation of mixtures of calcium nitrate with other salts is already known. Thus potassium or ammonium sulphate have 100 been mixed with calcium nitrate, the sulphates reacting with the calcium nitrate to form insoluble calcium sulphate and the corresponding nitrates. It has also already been proposed to introduce burnt lime with the object of forming a basic nitrate. The preparation of pro- 105 ducts of this kind, in which the calcium

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nitrate has been converted into other substances, does not come into consideration in the present case. Those salts which can give rise to undesirable reactions with calcium nitrate, are therefore to be added, after concentration of the calcium nitrate to 90—95% and before the cooling or rather, before or during the mechanical disaggregation at an elevated temperature.

It is already known to produce potash-containing mixed fertilisers by introducing potash salts such as potassium nitrate into calcium nitrate solutions. It has been shown that to the usual calcium nitrate in the molten form not more than 20% of potassium nitrate can be added, as otherwise masses result which are extremely tough and difficult to work with. In contradistinction to this the calcium nitrate-containing fertilisers obtainable by the present process can have incorporated with them any desired amounts of other fertilising salts, for example potash salts, in the manner indicated. The products thus obtained are quite crumbly, always set rapidly and are readily distributable and resistant to caking.

EXAMPLES.

1. 500 kg. of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ are melted in a kneading machine adapted to be heated and the melt further evaporated under vacuum (down to 100 m.m. mercury) to a content of calcium nitrate of 94% while the temperature is raised to 90° C. The melt, at first thinly liquid, becomes rapidly thick and immediately afterwards changes over to a sandy-pasty mass. This is cooled to 80° C. and can easily be brought by mechanical disaggregation at 80° C. into a granular form. The granules set very rapidly and become hard. The nitrogen content of the product is 16.0%.

2. 440 kg. of acetylene mud which contains 27.1% CaO , 0.2% MgO , 1% Fe_2O_3 and Al_2O_3 and 1.25% SiO_2 is treated with so much 50% nitric acid (about 560 kg.) that a neutral solution results. This is evaporated to a calcium nitrate content of about 75% then placed in a heated kneading machine and there further concentrated in vacuum to a content of over about 90% $\text{Ca}(\text{NO}_3)_2$. The temperature was increased to 110° C. thereby there quickly results a viscous product and shortly afterwards a sandy-pasty mass. This evaporated mass is then allowed to cool to 80° C. At this temperature it is capable by mechanical disaggregation of being converted into the granular form. The product so obtained solidifies rapidly

to compact, very hard granules. It contains 15.4% nitrogen.

3. 400 kg. of technical calcium nitrate solution (obtained by dissolving limestone in nitric acid) which contains 7.8% of nitrogen is evaporated in a kneading device until a sandy-pasty mass results which contains 15.5% of nitrogen corresponding to about 90% $\text{Ca}(\text{NO}_3)_2$. To this is added at 110° C., 200 kg. of potassium nitrate and the whole thoroughly kneaded. The mass is then cooled to 75° C. and can be easily converted into the granular form by mechanical disaggregation at this temperature, which on further cooling yields well solidifying hard granules. The fertiliser has a content of 23% K_2O and 14.6% N.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. Process for the preparation of strewable, non-caking fertilisers containing calcium nitrate in which calcium nitrate solutions are evaporated to a sandy-pasty consistency in which they contain about 90—95 of calcium nitrate, and thereupon the thus obtained product, without any further substantial concentration, is converted into a granular form by mechanical disaggregation in bulk at a temperature substantially above atmospheric of the order of 50° to 100° C.

2. Process as claimed in claim 1 in which the evaporation process for preparation of the 90—95 per cent. calcium nitrate product, preferably in its end phase, is carried out in vacuo in a kneading machine.

3. Process as claimed in any of the preceding claims in which other fertilising salts, preferably potash salts such as potassium nitrate, potassium chloride or potassium phosphate or mixtures of these, are added to the calcium nitrate, such salts as give rise to undesirable reactions with calcium nitrate being added after concentration of the original solution to about 90—95% calcium nitrate content.

4. Process for the production of a readily distributable and non-caking fertilizer containing calcium nitrate, substantially as described with reference to the accompanying examples.

5. Fertilisers whenever prepared or produced by the methods claimed.

Dated this 15th day of October, 1931.

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